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I, JULIE BILLINGSLEY, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2002951407 for a patent by WMC RESOURCES LTD as filed on 16 September 2002.

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Twenty-fifth day of September 2003

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Patents Act 1990

PROVISIONAL SPECIFICATION

Applicant(s):

WMC RESOURCES LTD

Invention Title:

IMPROVED RECOVERY OF VALUABLE METALS

The invention is described in the following statement:

IMPROVED RECOVERY OF VALUABLE METALS

The present invention relates to a process for improving recovery of valuable metals from iron-containing sulphides in ores and concentrates.

In many parts of the world, valuable metals such as gold, nickel and platinum group metals occur in iron-containing sulphides such as pentlandite, pyrrhotite and arsenopyrite. These minerals are recovered selectively from the ores by flotation.

While flotation is an efficient process usually, one of its most significant limitations for iron-containing sulphides is that fine particles are not recovered efficiently and a great deal of fine valuable sulphides are lost to the tailings.

US patent 6,170,669 (Senior et al) discloses a flotation process for iron-containing sulphide containing ores that is based on a finding of the inventors that a major reason that iron-containing sulphides float poorly at fine sizes (ie less than 130 micron) is that their surfaces are covered by an iron hydroxide film. This film prevents collector adsorption and renders the particles of iron-containing sulphides hydrophilic and poorly floatable with conventional sulphide flotation reagents.

The US patent discloses that the inventors believe that iron hydroxide films that are present in iron-containing sulphides consist of ferric hydroxide. The basis of the flotation process disclosed in the US patent is to strip the surface film for a time that is sufficient to allow collectors to adsorb onto the fine particles.

Specifically, the US patent discloses a flotation

process for iron containing sulphides in ores that includes adding a reducing agent, such as an oxy-sulphur compound (preferably dithionite), to an aqueous pulp of an ore that contains iron-containing sulphides at a pH of 7-10. Addition of the reducing agent decreases the pulp potential and thereby reduces and solubilizes an iron hydroxide film on the surface of iron-containing sulphides. The flotation process also includes adding a collector to the pulp and thereafter aerating the pulp to increase the pulp potential to a level sufficient to allow collector adsorption to the sulphides and subsequent flotation of iron-containing sulphides. The floated iron-containing sulphides can then be treated as required to recover valuable metals, such as gold, nickel or platinum group metals.

The flotation process disclosed in the US patent is based on laboratory flotation test work that was carried out on a batch basis.

The applicant has carried out research and development work on the flotation process disclosed in the US patent to develop the process for industrial application for recovering nickel from iron-containing sulphide ores.

The outcome of the further research and development work is a continuous conditioning process and apparatus for application of the conditioning process for subsequent flotation.

The present invention provides a continuous flotation process for iron-containing sulphides in ores in order to facilitate recovery of valuable metals from the iron-containing sulphides.

The method includes continuous conditioning and

flotation of an aqueous pulp of ores containing iron-containing sulphides.

In general terms, the method includes the following sequence of treatment steps on the pulp:

(a) adjusting the pH of the aqueous pulp to be in the range of 6.5-8.5;

(b) adding a reducing agent to modify an iron hydroxide film on the surface of iron-containing sulphides in the ores to enable collector adsorption onto iron-containing sulphides;

(c) adding a collector to the pulp prior to, during, or after adding the reducing agent in step (b); and

(d) aerating the pulp to increase the pulp potential to a level sufficient to allow collector adsorption onto the iron-containing sulphides;

(e) bubbling gas through the pulp and thereby subjecting the aqueous pulp to froth flotation to produce a froth containing said sulphide containing minerals.

Steps (a) to (d) are the conditioning steps and step (e) is the traditional flotation step.

The process also includes either one or both of the following steps.

One step is controlling the addition of the reducing agent in step (b) by reference to the change in

pulp potential as the reducing agent is being added.

The other step is adding a complexant to the pulp prior to or during step (b) of adding the reducing agent to react with the reduced iron produced in step (b) to minimise iron hydroxide reforming a film on the iron-containing sulphides.

Preferably the iron-containing sulphides contain one or more valuable metals selected from the group that includes nickel, gold, or platinum group metals.

Preferably step (a) includes adjusting the pH of the aqueous pulp to be in the range of 7.0-8.0.

Preferably step (a) includes adjusting pulp pH by adding acid to the pulp.

Preferably the acid is sulphuric acid.

Preferably step (a) includes adjusting pulp pH as the pulp flows through a first tank.

More preferably step (a) includes controlling the flow of pulp through the first tank to be plug flow.

Preferably step (b) includes controlling the addition of the reducing agent by adding the reducing agent to the pulp to decrease the pulp potential by at least 200 mV.

Preferably step (b) includes intensively mixing the pulp while adding the reducing agent.

Preferably step (b) includes adding the reducing agent to the pulp as the pulp flows through a second tank.

Preferably the reducing agent is an oxy-sulphur compound which dissociates in the aqueous media to form oxy-sulphur ions having the general formula

$$S_nO_Y^{z-}$$

where n is greater than 1; y is greater than 2; and z is the valance of the ion.

Preferably the oxy-sulphur compound is dithionite.

Preferably the complexant is selected from the group that includes citric acid and oxalic acid.

More preferably the complexant is citric acid.

Preferably the process includes adding the complexant to the pulp during step (b) of adding the reducing agent to the pulp.

More preferably the process includes adding the complexant to the pulp during step (b) of adding the reducing agent to the pulp as the pulp flows through the second tank.

Preferably step (c) includes adding collector that is selected from the group that includes xanthates, dixanthogen, xanthate esters, dithiophosphates, dithiocarbamates, thionocarbamates, and mercaptans.

More preferably the collector is a xanthate.

Preferably step (c) includes adding the collector to the pulp during step (b) of adding the reducing agent to the pulp.

More preferably step (c) includes adding the collector to the pulp during step (b) of adding the reducing agent to the pulp as the pulp flows through the second tank.

Preferably the process includes controlling the process so that the average residence time of pulp flowing through the second tank is less than 45 seconds.

More preferably the process includes controlling the process so that the average residence time of pulp flowing through the second tank is approximately 30 seconds.

Preferably the process includes controlling the flow of pulp through the second tank to be plug flow.

Preferably step (d) includes aerating the pulp downstream of the second tank.

Preferably step (e) includes separating the froth of floated iron-containing sulphides from the pulp and thereafter recovering valuable metals from the froth.

According to the present invention there is also provided an apparatus for continuously conditioning iron-containing sulphides in ores in order to facilitate recovery of valuable metals from the flotable iron-containing sulphides, which apparatus includes:

(a) a first tank for adjusting the pH of an aqueous pulp of the ores containing iron-containing sulphides as the pulp flows through the tank, the first tank having an inlet for receiving a flow of pulp and an outlet for discharging a flow of pH-adjusted pulp;

(b) a second tank for adding a reducing agent and a collector to the pH-adjusted pulp as the pulp flows through the tank, the second tank having an inlet for receiving the flow of pH-adjusted pulp from the first tank, an outlet for discharging a flow of treated pulp from the second tank, a means for adding the reducing agent to the second tank, a means for adding the collector to the second tank, and a means for intensively mixing the pulp in the tank; and

(c) a means for aerating the treated pulp from the second tank to allow adsorption of the collector onto the iron-containing sulphides surface.

Preferably the second tank also includes a means for adding a complexant into the second tank.

Preferably the inlet for the pH-adjusted pulp is in the lower section of the second tank and the outlet for treated pulp is in an upper section of the second tank.

With this arrangement, preferably the second tank includes a partition that divides the tank into a lower chamber and an upper chamber and the partition has a central opening that allows flow of pulp between the chambers. The purpose of the partition is to promote upward plug flow of pulp through the second tank.

Preferably the means for adding the reducing agent, the collector, and the complexant to the second tank are adapted to add these reagents to the lower chamber, whereby in use there is thorough mixing of the

pulp and the reagents in the lower chamber and plug flow of the pulp and the reagents upwardly through the chambers.

Preferably the first tank includes a means for venting air from the tank.

Preferably the means for aerating the pulp from the second tank includes a launder having an aeration screen located at the outlet of the second tank.

The present invention is described further by reference to the accompanying drawing which is a schematic diagram of a preferred embodiment of a flotation apparatus in accordance with the present invention.

As is indicated above, the process and apparatus of the present invention were invented in the course of research and development work carried out by the applicant.

The focus of the work was to improve recovery of nickel from fines and slimes streams in a flotation plant for iron-containing nickel-bearing ores.

The work investigated the potential application of the laboratory-based findings of the US patent to the flotation plant.

The work established that the flotation process disclosed in the US patent:

(a) could deliver improved recovery of nickel from the targeted fines and slimes streams in the flotation plant; and

(b) could be simplified to improve process

operation, reduce reagent costs, increase time of effect, and be operated on a continuous basis.

The work established that important factors in operating the flotation process on a continuous basis include:

(a) using change in pulp potential as a parameter for controlling the addition of reducing agent (such as dithionite) - this is a different approach to using absolute pulp potential as proposed in the US patent;

(b) the addition of citric acid leads to increased recovery benefit and increases the lasting effect of the process;

(c) using a specific design of apparatus that is based on two separate tanks to reduce reagent consumptions via minimisation of entrapped air consuming the reductant, low pH regions decomposing reductant, and increase process control by reducing pulp short-circuiting.

The specific design of apparatus developed in the work is shown in the drawing.

The apparatus includes a first tank 3 and a second tank 5 that are interconnected by a transfer pipe 7 extending between the lower sections of the tanks 3, 5.

The first tank 3 includes an inlet 15 for an aqueous slurry of pulp to be treated in the upper section of the tank 3 and the second tank includes an outlet for

treated pulp in the upper section of the second tank 5. The outlet is in the form of an overflow launder 17.

It can be appreciated from the drawing that pulp supplied to the inlet 15 of the first tank 3 flows downwardly through the tank 3 and from the tank 3 via the transfer pipe 7 and into the lower section of the second tank 5. Thereafter, the pulp flows upwardly through the second tank 5 and from the tank 5 via the overflow launder 17.

The second tank 5 is divided into a lower chamber 21 and an upper chamber 23 by means of a horizontal partition 25 that has a central opening that allows movement of pulp between the chambers.

The second tank 4 further includes a turbine 9 to generate turbulent mixing of pulp in the lower chamber 21 and an aerofoil impeller 11 to generate further mixing of pulp in a lower section of the upper chamber 23.

The second tank further includes baffles 18 to minimise the formation of a vortex and the subsequent entrainment of air into the tank that could result in decomposition of sodium dithionite.

The apparatus further includes a means for supplying reagents to the first and second tanks 3, 5. Specifically, the apparatus includes means for supplying acid (such as sulphuric acid) into the first tank 3, means for supplying a reducing agent (such as dithionite) into the second tank 5, means for supplying a collector (such as a xanthate) into the second tank 5, and means for supplying a complexant (such as citric acid) into the second tank 5.

The above-described arrangement of the two chambers 21, 23 defined by the horizontal partition 25,

the turbine 9, the impeller 11, and the means for supplying the reducing agent, conditioner and complexant to the lower chamber 21 promote plug flow of pulp upwardly through the second tank 5 with thorough mixing of the pulp and the reagents in the lower chamber 21 and sufficient reaction time for the pulp and the reagents as the pulp moves upwardly through the lower chamber 21 and the upper chamber 23 with minimal risk of short-circuiting of pulp/reactants.

The apparatus further includes a means for aerating pulp upon exiting the second tank in the form of an aeration screen 13 in the overflow launder 17.

The research and development work mentioned above investigated Slimes Rougher Tail within the WMC Mount Keith flotation circuit.

The following three steps were used in the work.

- First, the *Stage Recovery Improvement* was determined using comparative laboratory tests. This testing was conducted over a range of ore types.

- Second, *Cleaner Stage Evaluation* was undertaken using comparative laboratory tests. Partial deletion of sentence

- Third, *Pilot Plant Testwork* was conducted to assess scale up from laboratory to pilot plant and allow prediction of full scale plant performance. This phase of the work allowed confirmation of reagent consumption in continuous operation.

Stage Recovery Improvement - Laboratory Results

In brief, the process, hereinafter referred to as the "Fine Float Process" involves the following four steps:

- Addition of sulphuric acid to pH 7.3.
- Addition of citric acid and xanthate.
- Addition of sodium dithionite to decrease the pulp potential by 200mV.
- Aeration of pulp to increase the pulp potential.

Baseline laboratory flotation tests on column tailings resulted in an average Ni recovery of 48.0% at a grade of 1.0% from a feed grade of 0.30%Ni.

Thirty-six paired laboratory tests were conducted to determine the effect of applying the Fine Float Process on the Ni recovery and grade. The comparative tests were conducted over a range of ore types presented to the plant. Each Fine Float Process test was compared to a corresponding baseline test with the difference in Ni recovery and grade determined. The average Ni recovery and grade benefit of 36 paired tests, associated with application of the Fine Float Process, was 11.7%(±1.5%) and 0.2%(±0.6%), respectively.

Cleaner Stage Evaluation

Rougher-Cleaner tests indicated that one application of the Fine Float Process to Slimes Rougher Tail increased recovery by approximately 10% from 55.9% to 66.1%. Subsequent cleaner flotation of that concentrate resulted in a cleaner stage recovery increase of 13.6%

from 70.8% to 84.4%. The overall recovery increased from 39.6% to 55.8% at concentrate grades of 4.4% and 7.9% respectively.

Pilot Plant Testwork

A pilot plant was used to evaluate scale-up of the Fine Float Process form laboratory to pilot plant and to allow prediction of full scale plant performance. A scaled version of the conditioning vessel was used in this testwork and reagent addition was controlled automatically to pulp potential and pH. The Slimes Rougher Tail stream was also selected for this work.

The recovery and grade benefit obtained in pilot plant testing for 7 ore types (32 paired tests), via application of the Fine Float Process, was 9.7%(±3.7%) at an average Ni grade increase of 0.0%(±0.1%) as seen in Table 1 below.

TABLE 1

Comparison of results in pilot plant with and without Fine Float Process

	Ni Recovery %	Ni Grade %
Baseline	45.4	0.76
Fine Float Process	55.1	0.76

In summary:

- Application of the Fine Float Process in laboratory work had significantly increased stage recovery of slimes particles over a range of ore types.

- In addition to a rougher stage improvement, the Fine Float Process was found to impart a sustained recovery improvement in subsequent flotation stages.

- The laboratory results were scaled to a continuous pilot plant increasing confidence in predicting plant performance. The conditioning vessel provided a means of applying and controlling the chemical process.

Many modifications may be made to the embodiments of the process and apparatus of the present invention described above without departing from the spirit and scope of the invention.

Fine Ni Conditioning Vessel